INVESTIGATION OF THE AMMONIUM PERCHLORATE COMPOSITE PROPELLANT DEFLAGRATION MECHANISM BY MEANS OF EXPERIMENTAL ANALOG TECHNIQUES

Technical Report ME - RT 67006

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Robert F. McAlevy, III, Suh Yong Lee, Frank A. Lastrina, and Norman A. Samurin

September 1967

Prepared for

DEPARTMENT OF THE NAVY
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Contract Nonr 263 (48)





# STEVENS INSTITUTE OF TECHNOLOGY

DEPARTMENT OF MECHANICAL ENGINEERING

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# INVESTIGATION OF THE AMMONIUM PERCHLORATE COMPOSITE PROPELLANT DEFLAGRATION MECHANISM BY MEANS OF EXPERIMENTAL ANALOG TECHNIQUES

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Stevens Institute of Technology
Department of Mechanical Engineering
Hoboken, New Jersey

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#### ABSTRACT

Laboratory burners have been employed as a means of studying experimentally, by analogy, the ammonium perchlorate (AP) composite propellant deflagration process. ber of theoretical models of the propellant deflagration process have been proposed. However, structural peculiarities of composite propellants prevent gathering the combustion data required for their evaluation. Two types of experimental analogs have been developed and employed for this purpose. One type, the "porous-plug" burner, involves the use of a porous AP bed through which fuel gases are passed and burned at the regressing surface; similarly, oxidant gases are burned with porous thermoplastic beds. The other type, the "loose-granule" burner, involves mixtures of solid AP and thermoplastic granules that are burned without a gaseous component. Thus, it is possible to evaluate the effects of fuel and oxidant initial physical phase as they enter the flame zone, system chemistry, and the scale of granularity (of both fuel and oxidant) on the deflagration process.

Comparison of the loose-granule burner deflagration characteristics with those of actual composite propellants demonstrated that the technique is an excellent, if not perfect, analog and that the propellant thermoplastic fuel-binder pocket size might play an unexpectedly powerful role

in the propellant deflagration process.

Comparison of "loose-granule" and "porous-plug" burner characteristics provided additional support to the view that the details of energy release rate distribution patterns have an influence on the deflagration rate of systems containing polystyrene and polymethylmethacrylate. Thus, such rates cannot be realistically described only in terms of overall energy release as suggested by others.

The limits of both burners as analog techniques have been established. For combustion pressures greater than 35 psia or so, depending on system granularity, flame pener tration into the interstices between granules leads to unstable combustion of the loose-granule burner and destroys its analogy to normal composite propellant deflagration. The bonding technique employed in porous-plug burner fabrication significantly influences its combustion characteristics and thus mitigates the inferences that might be extracted from them. This suggests the work of others in determining stolchiometrically correct mixture ratios from burning rate-mixture ratio characteristics should be re-examined.

The subject results reinforce the view that the burner analog technique is the best means currently available for testing the validity of proposed models of the ammonium perchlorate composite propellant deflagration mechanism.

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#### SECTION 1

#### BACKGROUND AND INTRODUCTION

Ammonium perchlorate (AP) composite propellants are widely employed in currently operational solid rocket motors, as well as those programmed for future development. The combustion mechanism of this type of propellant is unknown, and so it has been impossible to produce a rational basis for motor design, or even scaling rules for adapting successful motors to new applications. For example, the effect on combustion stability and efficiency, ease of combustion extinction, etc. of doubling the size and reducing to half the operating pressure of a proven motor cannot be predicted with reasonable reliability. Consequently, extensive development testing is required for each new motor or new application. expensive, both in time and money, and there is no reason to believe that it will always be successful. Therefore, it appears that the future growth of solid propellant rocket technology may well be paced by the speed with which the propellant deflagration process can be elucidated.

Composite solid propellants usually consist of granules of ammonium perchlorate, metallic powder (such as beryllium or aluminum), polymeric binder, plasticizer and

curing agent. These components are mixed together to obtain a heterogeneous mixture, after which they are cast into motor casings. During casting, care is taken to insure a solid integral mass results, thereby making certain that no inclusions occur due to trapped gases which may cause structural weakness. The cast mixtures are then placed in a controlled temperature oven for, typically, 72 to 196 hours at 150 to 190 °F for curing. At all times extreme care must be taken with these high energy materials so as to avoid detonations or explosions.

The burning surface of such propellants reflects the heterogeneity of their compositions; they are composed of a complex of randomly scattered oxidizer and metallic particles, separated only by as much binder as is necessary to fluidize the mix before curing and to produce desired mechanical properties in the propellant. (Nevertheless, in order to produce practical propellants of stoichiometrically correct mixture ratio, oxidizer crystals in at least two different size ranges normally must be employed.) As each surface layer burns away new particles randomly emerge to replace those being consumed; therefore, the structure of the surface and the advancing flame that consumes the vapors emerging from the surface are both time dependent.

This formidable geometric factor has stymied the

development of any complete theoretical analysis of the practical composite solid propellant deflagration process. (Of course, any such analysis would have to account for the normal combustion complexities of chemical reaction kinetics and transport phenomena indigenous to all flames; and, in addition, include a description of the surface vaporization process - which, at the present time is unknown for the propellant ingredients of interest.) lieu of this, various investigators have suggested different steps in the deflagration process as "controlling" the overall rate in order to produce simplified models that are susceptible to analysis. Typically, these heuristic approaches have resulted in algebraic relationships between deflagration rate and: combustion pressure, oxidant-fuel mixture ratio, ammonium perchlorate crystal size, chemical nature of the fuel-binder, etc. To date, this has been done explicitly only for metalfree systems based on ammonium perchlorate crystals of a single size.

The next step in the development of an understanding of the deflagration process on a level that will be useful for engineering purposes is to determine which of these models best represents the actual process, if, indeed, any can be gainfully employed in this way. A means for doing so, and, in fact, for generating improved models,

is the subject of an extensive research program under way in the Combustion Laboratory, and this document reports some recent results.

### A. Propellant Deflagration Models

Reviews of the current status of the field have been presented recently by Barrère and Williams (Ref.1) and by R. Friedman, et al. (Ref.2). These reports contain a comprehensive list of references to the pertinent work of the past, and they are commended.

The simplistic models that have been postulated result from sweeping assumptions concerning the actual deflagration process, e.g., that both quasi-steady and quasi-one-dimensional representations are valid.

All of the models that have been given serious consideration are thermal in nature, e.g., the surface regression rate is related directly to the rate of thermal energy feedback from the flame above. Thus, by incorporating a heat sink at the cold boundary to account for the phase change (and heat source to account for chemical heat generation), and including a mechanism for material transport normal to the direction of principal energy flux, recourse can be made to the much-studied thermal theory of premixed gaseous flame propagation. (It is appropriate to mention here that as a result of several years of quite

productive effort, Hirschfelder (Ref.3) and his group successfully formulated, in complete generality, the equations governing the propagation of a premixed, gaseous, laminar flame. However, despite a massive attack, they were unable to solve completely the equations for any real flame because of mathematical complexities.)

When only the conductive mode of energy transfer back to the surface is considered, the flame thickness, "t", and flame temperature, T<sub>f</sub>, are of paramount importance in establishing the surface heat flux level. The linearized relationship between energy absorption rate at the surface and energy heat flux to the surface can be expressed, in the case of conductive heating, as:

$$\lambda_{\text{eff}} \frac{(T_{\text{f}} - T_{\text{s}})}{t} = \dot{m} q_{\text{eff}}$$
 (1)

where:  $\lambda$ eff is the effective thermal conductivity of the flame

T<sub>s</sub> is the temperature of the propellant surface

m is the mass consumption rate per unit area

qeff is the effective heat of vaporization per unit mass.

With propellant chemistry constant,  $T_f$  and "t" are both subject to experimental control:  $T_f$ , through its dependence on oxidant-fuel mixture ratio, and "t", through its dependence on combustion pressure, propellant

scale of granularity and mixture ratio. The resulting variation of m can be measured quite readily. (The quantity usually measured is the propellant surface regression rate, r, which is m divided by the propellant density.) Therefore, it appears that the validity of Eq. 1 in describing the composite propellant deflagration processes could be easily verified experimentally; however, this is not possible by means of experiments performed with the propellants themselves because the significant parameters cannot be varied independently.

For example, although propellant granularity is usually characterized by oxidant granule size,  $\delta_{\rm o}$ , "t" might depend strongly on the "granularity" of the pockets of fuel-binder between the oxidant granules,  $\delta_{\rm f}$ . In order to construct a realistic model of the deflagration mechanism it is necessary to determine the relative importance of  $\delta_{\rm f}$  and  $\delta_{\rm o}$  in establishing "t". But when  $\delta_{\rm o}$  is held constant and more fuel-binder is added to increase  $\delta_{\rm f}$ , then T<sub>f</sub> is changed as well (since the mixture ratio is changed). On the other hand, when T<sub>f</sub> is held constant,  $\delta_{\rm f}$  can be varied only by varying  $\delta_{\rm o}$ . Fig.1 depicts schematically the variation of  $\delta_{\rm f}$  with  $\delta_{\rm o}$  and mixture ratio. It is clear that  $\delta_{\rm f}$ ,  $\delta_{\rm o}$  and T<sub>f</sub> cannot be varied independently with actual composite propellants. Further, for propellants incorporating

oxidants of a single crystal size, that is, the only type of propellant that has been treated theoretically, only fuel-rich compositions can be manufactured by normal casting precedures; the range of  $T_f$  variation between this casting limit and the fuel-rich combustion limit is quite narrow for such propellants, and, therefore, so is the variation in  $T_f$  achievable by mixture ratio variation.

Consequently, the validity of theories of the composite propellant deflagration process based on heat conduction from the flame to the surface cannot be established on the basis of the measured response of r to variation of experimental parameters.

A number of assumed variations of "t" on combustion pressure, P, have resulted in various predictions of r on P (Refs.l and 2) which have had various degrees of success in correlating propellant r (P) data. However, successful correlation of such gross experimental data is a necessary, but certainly not a sufficient, condition for establishing the validity of a proposed deflagration model. The assumed model underlying the analysis must be tested directly, and from the preceding discussion it is clear that such tests cannot be made with actual composite propellants. Other means must be used.

Experiments have been made with laboratory burners that were employed as simulators of the actual propellant

deflagration process. The validity of the analogs will be established by comparing the deflagration characteristics of the burners to those of composite propellants. The means of comparison will be an equation produced by Summerfield (Ref.4). Before proceeding with this comparison the ability of the Summerfield Equation to represent propellant deflagration data must be established.

### B. Test of the Summerfield Equation with Propellants

An assumed variation (Ref. 4) of "t" inserted into Eq. 1 resulted in

$$\frac{P}{r} = a + bP^{2/3} \tag{2}$$

where: "a" is a "reaction time" parameter, depending strongly on  $T_f$ 

"b" is a "diffusion time" parameter, depending strongly on propellant scale of granularity.

Within the framework of the model the reaction time parameter is independent of \$\int\_{\text{o}}\$ and P, and is a function only of propellant chemistry; and the diffusion time parameter is a function of granularity and not of pressure. A proposed dependence of "b" on granularity has been suggested (Ref.5) as b = b' \$\int\_{\text{o}}\$ where "b'" is assumed to be independent of both pressure and granularity. Substituting this relationship into Eq. 2 yields

$$\frac{\mathbf{p}}{\mathbf{r}} = \mathbf{a} + \mathbf{b}^{\mathsf{T}} \mathbf{\delta}^{\mathsf{2}/3}, \tag{3}$$

an equation which allows the possibility of correlating the parameters  $\boldsymbol{\delta}$  and P with r at constant mixture ratio in two different planes, the P/r - P<sup>2/3</sup> plane and the P/r -  $\boldsymbol{\delta}_{0}$  plane. Bastress (Ref.5) tested a number of composite solid propellants (ammonium perchlorate in a polysulfide fuel-binder) of varying mixture ratio and oxidant granule size at different pressures; these data can be viewed within either of the aforementioned planes.

Bastross' data when plotted in the P/r -  $P^{2/3}$  plane could be described, within a limited range of pressure and granule size, by a series of straight lines as predicted by Eq. 2, with "b" the slope and "a" the intercept. Fig.2 displays some of the data (Ref.5) for which the straight line prediction holds, e.g., for  $15 \leq P \leq 50$  psia and  $78 \leq 6 \leq 230 \mu$ . (As the granules employed in each propellant were not all the same size,  $6 \leq 100$  is a number representative of the average granule diameter.) The reaction time parameter, "a", is not constant as assumed by the equation, but a function of  $6 \leq 100$ . The assumed  $6 \leq 100$ 0 dependence of "b" is not completely consistent with the experimental results, since the plot of b vs  $6 \leq 100$ 0 for the data of Ref.5, depicted in Fig.3, while linear, does not extrapolate to "b" equal zero for  $6 \leq 100$ 0 equal zero.

Values of "b" extracted from the data of Ref.5 are tabulated in Table 1.

The data of Fig.2 replotted in the P/r -  $\delta_{\rm O}$  plane at constant pressure, Fig.4, can also be described by a series of straight lines as predicted by Eq. 3. Extrapolating these lines to  $\delta_{\rm O}$  equal zero, however, indicates that "a" is also a function of pressure contrary to this theory. These values of "a", determined by varying  $\delta_{\rm O}$  while holding P constant, and those obtained by varying P while holding  $\delta_{\rm O}$  constant (as described previously) are tabulated in Tables 1 and 2.

As predicted by Eq. 3, the straight line correlations of Fig.2 have slopes of b' \( \begin{align\*}{6} \) and those of Fig.4 have slopes of b' \( \begin{align\*}{6} \) For each of these straight lines a "b'" can be extracted. If the Summerfield scheme resulted in a precise representation of propellant data, all such values of b' should be the same. However, as can be seen from Tables 1 and 2 these values are not the same and, therefore, b' is a function of both pressure and granularity.

Clearly the assumed model, while apparently representing some aspects of actual propellant deflagration characteristics, misrepresents others. Hence, comparisons between actual propellant combustion characteristics and those of the burner analogs based on this scheme will be uncertain to a degree that reflects this misrepresentation.

### C. Propellant Deflagration Analog Techniques

Various types of analog techniques have been employed to model experimentally some aspect or aspects of the composite propellant deflagration process (Refs. 1,2,6,7,8,9,10,13 and 14). One such propellant analog is the pressed strand burner, (Refs. 13 and 14), composed of granules of AP and various types of fuels compacted to form a propellant strand. Two other analog techniques, that do not involve compaction in their fabrication, have been developed in the Combustion Laboratory; these appear to model the essential elements of the process, including the detailed structure of the flame zone and the burning surface, while still permitting ready experimental control of sensitive parameters over a much broader range than is possible with actual propellants.

One involves the use of a porous-plug in which gaseous fuel is passed through a burner tube laden with point -of-contact bonded ammonium perchlorate granules (or, alternatively, in which gaseous oxygen is passed through a burner tube laden with point-of-contact bonded thermoplastic granules). The other involves the use of a burner laden with an unbonded mixture of ammonium perchlorate

and thermoplastic granules.

Results obtained with the porous-plug burner when compared to those of the loose-granule burner have already produced a new insight concerning the nature of the surface boundary condition for ammonium perchlorate combus-Ammonium perchlorate crystals were grown on thermocouples, and then incorporated in both types of burners; the surface temperatures of each type of burner were found to have values that were quite different, although the combustion pressures and surface regression rates were approximately the same (Ref.6). This finding upset both of the then-prevalent views of the oxidant combustion boundary condition (i.e., thermodynamic equilibrium vs. chemical kinetically "controlled"), and have led Powling to propose a new view of this boundary condition for the combustion of actual composite propellants (Ref.11). is cited to illustrate the powerful impact burner analog studies already have had on the development of composite propellant deflagration models.

The usefulness of the porous-plug burner has been reported upon previously (Refs. 6,7, and 8). Briefly, its principal advantage stems from the fact that the experimenter selects the rate at which either the fuel or oxidant is fed into the flame zone. In propellants, both components vaporize at a rate that is fettered to the rate

at which heat is fed back to the surface. The porous-plug burner permits variation of both fuel and oxidant "pyrolysis" rates and mixture ratio over a much broader range than is possible with actual propellants.

The loose-granule burner permits the experimenter to vary independently  $\mathbf{S}_{0}$ ,  $\mathbf{S}_{f}$  and  $\mathbf{T}_{f}$  (through its dependence on mixture ratio). As described above, these are fettered together in composite propellants due to their peculiar structure.

This document reports on further studies of ammonium perchlorate composite propellant deflagration by means of these burner analog techniques. The region of applicability of each has been delineated. The influence of ammonium perchlorate porous-plug burner fabrication technique on exhibited deflagration characteristics has been studied. And the validity of the loose-granule burner analog has been established by comparison of its combustion characteristics with those of actual composite propellants via the Summerfield scheme.

#### SECTION II

#### EXPERIMENTAL TECHNIQUE

Two types of burners have been developed in this laboratory as experimental analogs of the composite propellant deflagration process: the porous-plug burner and the loose-granular burner. The porous-plug burner is composed of either fuel or oxidizer granules packed into a burner tube and point-of-contact bonded. The burner tubes are either of steel (in which case timing wires are employed for surface regression rate measurement) or of glass for visual observation of the regressing surface. The loose-granule burner is composed of a mixture of oxidizer and fuel granules packed into a burner tube of either steel or glass. The details of the fabrication of these burners are in Appendix A.

For control of the burner combustion pressure two pressure housings have been constructed within which either type of burner can be mounted. The original housing (Ref.7) permitted regression rate data to be obtained by means of the standard timing wire technique. A "modified" pressure housing was constructed to permit optical study of the combustion process, especially the nature of the regressing surface. In order to keep the observation windows clean, the modified housing provided for the

continuous removal of combustion gases. Figs. 5 and 6 are schematics of the original and the modified housings respectively with their support equipment. Details of the original housing are in Ref. 7 and those of the modified housing are in Appendix B.

The details of the electrical circuitry employed in regression rate measurement by the standard timing-wire technique are presented in detail in Appendix C. The use of 3 timing wires with each burner permitted 2 measurements of regression rate for each run.

The materials used for this investigation--ammonium perchlorate (AP), polystyrene (PS), and polymethylmethacrylate (PMM)--are described in Appendix D, as is the method of particle size classification.

#### SECTION III

#### RESULTS AND CONCLUSIONS

# A. Effect of Fuel Combustion Characteristics on Burner Deflagration

Two thermoplastics, polystyrene and polymethylmethacrylate, typical of those employed in composite propellants were previously tested in the porous-plug burner with gaseous  $0_2$  as the oxidant (Ref.6). During the subject investigation these thermoplastics were studied in the loose-granule burner with AP granules as the oxidant. Plotted in Fig. 7 is the measured burner deflagration rate, r , as a function of equivalence ratio,  $\Phi_{\text{c/f}}$  , for both analog techniques (Equivalence ratio is defined here as the measured oxidant-fuel consumption ratio normalized by the stoichiometrically correct ratio; the latter is computed by assuming that the fuel is burned to  $CO_2$  and  $H_2O$ , and O2 is the only active oxidant and that ammonium perchlorate decomposes as follows:  $NH_4ClO_4 \rightarrow 1.25 O_2 + 1.5$  $H_2O + HCl + 0.5 N_2$ ). Each point of Fig. 7 depicts the arithmetic mean of at least 3 runs; all data obtained lie within +3 percent of these arithmetic mean points. tests were performed at atmospheric conditions.

The porous-plug burner data depicted in Fig. 7 show

that the burning rates of the PS system are greater than those of the PMM system. It has been hypothesized (Ref.6) that this is probably a result of different energy distribution patterns throughout the flame zones of the two That is, since they both have about the same values of " $T_f$ " and " $q_{eff}$ " (Ref.12), the differences in "r" probably stem from differences in "t" teresting to note that similar differences between the two systems are exhibited in practical propulsion systems; for example, in a conventional hybrid rocket motor the PS - 0, system produced about twice the burning rate of the PMM - $O_2$  system (Ref.12). The porous-plug r vs  $\mathbf{E}_{O/f}$  characteristics for PS and PMM peak at  $\frac{\mathbf{\Phi}}{\mathbf{O}/\mathbf{f}} = 0.5$  and  $\frac{\mathbf{\Phi}}{\mathbf{O}/\mathbf{f}} = 1.0$ respectively (Fig. 7). This finding coupled with the above hypothesis has led the authors of Ref. 6 to conclude that PS burns in a two-stage flame, while PMM burns in a onestage flame, when O is employed as the oxidant. Recently, Russian researchers (Ref.13) came to the same conclusion based on their observations of the pressed strand burner combustion characteristics.

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The loose-granule burner data of Fig. 7 also exhibit burning rate maxima, though, they are less pronounced. Therefore, the aforementioned conclusion concerning PS vis-a-vis PMM flame zone structure can be drawn for the loose-granule burner with solid AP as the oxidant as well

as for the porous-plug burner with gaseous 0, as the oxidant. Detailed interpretation of the data was prevented by the different granularities of the materials employed, a result of insufficient supplies at the time of the experiments. Nevertheless, the results obtained indicate that the fuel nature has an influence on the combustion characteristics of systems employing solid AP (as well as gaseous  $0_2$ ) as the oxidant over and above that of its overall heat of combustion. This influence must be accounted for in any complete model of the deflagration mechanism of such systems if it is to be realistic. conclusion is contrary to the contention of Powling (Ref. 11) that the burning rates of composite mixtures of ammonium perchlorate with organic fuels are little affected by fuel-binder properties, other than heat of combus-It is consistent with results of Powling tion , at low pressures. Russian workers (Ref. 13) based on their observation of combustion characteristics at high pressures.

# B. Effect of Porous-Plug Fabrication Technique on Burner Deflagration

Ammonium perchlorate porous-plug burners have been employed previously in the Combustion Laboratory (Refs. 6, 7 and 8) and in the University of Louvain in Belgium

(Refs. 9 and 10). In both laboratories point-of-contact bonding of the AP plugs has been accomplished, but different types of solvents were employed. In the Combustion Laboratory the burners were bonded by using a saturated solution of AP in water; it was assumed that use of this bonding solution left the granule size essentially unaltered and that the final burner contained only AP. At the Louvain laboratory the burners were bonded using a solution of PMM in acetone so that the AP granules were cemented together by a thin coating of PMM.

Burners bonded by both techniques were tested during the subject program to assess: (1) the region of applicability of the AP-water solution bonded porous-plug fabricated in the Combustion Laboratory as a composite solid propellant deflagration analog and; (2) the similarities and differences between the two types of AP porous-plugs, especially with regard to the influence of the PMM cement on the deflagration characteristics.

### i Limitation of AP-water solution bonding

Fig. 8, a plot of r vs  $m_f/m_o$  at constant pressure levels, displays data obtained with AP burners fabricated with the AP-water solution. With r measured in the standard way, the measured AP loading density in the tube permitted calculation of  $m_o$  (Ref. 8). Each point depicts

the arithmetic mean of at least 3 runs; all data lie within  $\pm$  3 percent of these arithmetic mean points for the 15 psia tests.

At conditions to the right of the shaded line (Fig.8) visual observation disclosed individual AP granules leaving the plug surface and burning as they were carried downstream by the emerging methane (Fig. 9). Since surface regression was influenced by this phenomenon of AP granule dislodgement and downstream burning which does not normally occur during combustion of composite propellants, it is concluded that the subject type of porous-plug burner is not a good analog of the AP composite propellant deflagration process at operating conditions to the right of the shaded line. Thus, it appears that this phenomenon imposes a pressure-dependent upper limit on the mixture ratios for which the porous-plug burner comb e an effective analog to solid propellant deflagration processes.

Further studies of the limit of the AP porous-plug analog were conducted at atmospheric pressure in an effort to separate the effects of thermal-mechanical stress on granule dislodgement. One such study, designed to determine the effect of throughput rate on mechanical strength, was conducted by flowing gas through the porous-plug without combustion. Visual observation revealed no

AP leaving the surface for gas throughput rates even greater than that which produces the fuel-rich combustion limit.

The other study involved combustion of the porousplug. First, by means of inert diluents, Tf was varied at constant gas throughput rate, and second, gas throughput rate was varied at constant  $T_{f}$ . Depicted in Fig. 10 are data obtained during this study. Each point represents the arithmetic mean of 3 runs; all data lie within + 3 percent of the arithmetic mean points. At conditions to the right of the shaded areas visual observations disclosed individual AP granules leaving the plug surface and burning as they were carried downstream (as in Fig.9). Attempts to relate the boundary between burning with AP dislodgement and burning without AP dislodgement to variations in gas throughput and T, by means of a simple connection proved futile. And so it was not possible to separate the effects of thermal and mechanical stress in AP porous-plug burner breakup.

It was noticed that at operating conditions very near, but still to the left side of, the shaded line in Fig. 8 a flame appeared at the top of the burner tube. Such flames were similar to that of Fig. 9. It is assumed that this flame is a result of combustion of unburned methane with oxygen from the air within the housing.

This same condition, afterburning of methane at the top of the tube, also existed for the diluted methane throughputs. That is, this flame would appear at values of  $\mathfrak{m}_f/\mathfrak{m}_0$  to the left side of the shaded regions of each curve of Fig. 10. Assuming mixing is complete before emergence from the tube this flame can be considered to indicate fuel-rich combustion in the tube. Hence, stoichiometrically correct combustion is probably accomplished at mixture ratios lower than that at which the analog is limited owing to dislodgement of the granules.

# ii Effect of PMM-acetone solution bonding on deflagration rate

The influence of AP porous-plug bonding technique on burner deflagration characteristics was investigated with PMM-acetone solutions of varying strength. It was determined that 1.0 gm, 2.0 gm or 3.0 gm of PMM dissolved in 100 ml of acetone resulted in solutions that produced strong point-of-contact bonding of the porous-plug. However, the 1.0 gm PMM-acetone bonded porous-plug exhibited the same phenomenon as the above mentioned AP-water solution bonded porous-plug, i.e. granule dislodgement, but at a value of m greater than that of the AP-water solution bonded plug. This phenomenon was not observed for the 2.0 gm or 3.0 gm PMM-acetone bonded plugs.

These burners were tested at atmospheric pressure with methane throughputs. The results of the 3.0 gm PMM in 100 ml acetone are displayed and compared in Fig. 11 with those obtained with burners fabricated with the saturated AP-water solution. Many tests were run at each condition and the shaded areas are drawn to encompass extreme data points from each type of burner. For  $\dot{m}_f/\dot{m}_O$ greater than 0.05 the burners exhibit values of r that are within a few per cent of each other, although the dependence of r on  $\dot{m}_f/\dot{m}_O$  is somewhat different for each, and the value of  $\dot{\mathbf{m}}_{\mathbf{f}}/\dot{\mathbf{m}}_{\mathbf{O}}$  that produces a maximum value of r depends on the burner fabrication technique. for the burners fabricated by means of the PMM-acetone solution, it was found to depend on the PMM concentration -for 3 gm in 100 ml, the  $m_f/m_c$  for maximum r was 0.095, while for 2 gm in 100 ml, it was 0.075.

The workers at the University of Louvain (Ref. 10) used a 0.4 gm PMM in 100 ml acetone solution for bonding their porous-plugs and obtained satisfactory point-of-contact bonding. However, a 0.5 gm PMM in 100 ml acetone solution, when investigated by the authors, did not satisfactorily bond the porous-plug; the porous-plug granules became dislodged by gas throughput without combustion. Evidently the bonding solution is but one source of the displayed differences. Each step of the overall technique,

may be important and should be examined in order to ascertain the different results obtained.

Two porous-plug fabrication techniques were examined at the University of Louvain (Ref. 10). In comparing the data obtained with their samples bonded via the previously mentioned PMM-acetone solution to the data obtained by the porous-plug fabrication technique described in Ref. 7, they found an 8 per cent increase in burning rate due to PMM-addition, even though dislodged granules were observed with the Ref. 7 technique. However, because of the limited quantity of data obtained, they did not observe that the value of m<sub>f</sub>/m that produces a maximum value of r depends on the burner fabrication technique employed (as observed in this work - see Fig. 11). Since the mixture ratio at which the maximum burning rate occurs was employed at Louvain (Ref. 9) as a basis for determining the stoichiometry of the AP-methane system, as well as others, the stoichiometries they obtained must reflect an influence of the PMM cement.

Based on the above results, the following conclusions can be drawn:

(1) The afterburner flame, appearing at the top of the burner tube at  $\dot{m}_f/\dot{m}_O$  less than 0.085, indicates that the stoichiometrically correct value for the system must be

less than this value. Since the workers at the University of Louvain (Ref. 9) derived an AP decomposition scheme based on this value being the stoichiometrically correct value, deduced from the observation  $r = r_{max}$  at  $m_f/m_c = 0.085$  with the PMM-bonded burners, the validity of their conclusions are open to serious question.

- (2) The location of the measured maximum burning rate is influenced by the presence of foreign materials, in this case PMM cement.
- (3) For porous-plug burners, bonded using an AP-water solution it was impossible to obtain r at sufficiently fuel-rich conditions to discern accurately the value of  $\dot{m}_f/\dot{m}_O$  that produces a maximum r,
- (4) Therefore, determination of the AP combustion stoichiometry will probably require measurement of the overall chemical energy release and/or combustion product species concentration, since neither of the current porous-plug burner analog techniques can be employed for this purpose when r is the only independent measured variable.
- C. Effect of Mixture Ratio and Combustion Pressure on the Loose-Granule Burner Deflagration Characteristics

Shown in Fig. 12 is log r vs log P at constant  $m{\delta}_{\rm O}$  (500 - 420  $\mu$ ) and  $m{\delta}_{\rm f}$  (420 - 350  $\mu$ ) for three mixture

ratios of PS and AP. This plot indicates three deflagration regimes of the loose-granule burner. Regime I is a stable burning region where the data can be correlated well by the familiar power law relationship:

$$r = BP^{n}$$
 (4)

where: B is a constant for each system

n is a constant for each system

In Regime I, n <1, and data scatter is small (<+ 4 per cent over at least 3 runs), Regime II, a transition region, is characterized by large scatter in the data (>+ 4 per cent over at least 3 runs) and a distinct change in the pressure exponent from that of Regime I. Regime III is an unstable combustion region. In this regime the chamber pressure fluctuates during burning, and visual observations indicate propagation of the flame front into the interstices between granules below the regressing surface. Thus, it is concluded that this subsurface flame propagation limits the loose-granule burner analog in representing the deflagration characteristics of a composite solid propellant, since well-made propellants do not include interstices within which such subsurface flame propagation can occur.

The same general results were obtained with burners

fabricated with fuel granularities,  $\delta_{\rm f}$ , of 500-420 $\mu$ , 350-297 $\mu$  and 297-250 $\mu$ , and  $\delta_{\rm o}$  at 500-420 $\mu$ , when tested in the same pressure range. However, burners produced by holding constant the mixture ratio, 82% AP, and  $\delta_{\rm f}$ , 500-420 $\mu$ , while changing the oxidizer granularity,  $\delta_{\rm o}$ , (350-297 $\mu$ , 297-250  $\mu$ , 210-177  $\mu$  and 177-149 $\mu$ ) did not exhibit Regimes II and III when the pressure was varied between 16 to 51 psia. On the basis of these preliminary results it is provisionally concluded that the behavior characteristic of Regimes II and III is produced when  $\delta_{\rm f}$  /  $\delta_{\rm o}$   $\angle$  1. Further study will be required before this phenomenon can be understood.

D. Comparison of the Deflagration Characteristics of the
Loose-Granule Burner and Composite Solid Propellants
by Means of the Summerfield Equation

As described in the BACKGROUND AND INTRODUCTION the Summerfield Equation is a relationship between pressure, burning rate and granularity. Propellants have only one measurable granularity, that of the oxidant; the fuel "granularity" is inherently a function of the oxidizer granularity. In contrast, the loose-granule burner has two independent granularities, that of the oxidizer (  $\delta_{\rm o}$  ) and that of the fuel (  $\delta_{\rm f}$  ). This additional degree of freedom in the loose-granule system must be considered in

viewing loose-granule burner data in light of the Summerfield Equation. Thus loose-granule burner data may be viewed in an additional plane, P/r vs  $\delta_{\rm f}$ , as well as in the P/r vs P<sup>2/3</sup> and P/r vs  $\delta_{\rm o}$  planes, which, as previously pointed out, are of significance in interpreting propellant data. The following discussion is organized around comparative views of burner and propellant data in these planes.

# i Comparisons via the $P/r - P^{2/3}$ plane

The previously discussed data of Fig. 12 for which mixture ratio is varied have been replotted in the P/r vs P<sup>2/3</sup> plane, Fig. 13, to allow interpretation via the Summerfield Equation. Naturally, only data from Regime I, within which stable burning was observed, are so plotted. The linear correlation apparent for each of the three mixture ratios at constant granularity, demonstrates the applicability of the Summerfield Equation within the range of pressure variation employed. This linear correlation is in striking analogy with propellant data for different mixture ratios, as reported in Ref. 4 and Ref. 5; that is, propellant data and the loose-granule burner data both exhibit straight line correlations in this plane.

Data for three typical loose-granule burner formulations of the same mixture ratio but of different fuel (PS) and oxidant (AP) particle size combinations are presented in the P/r vs  $p^{2/3}$  plane of Fig. 14. The typical data shown, as well as all other data obtained in this investigation, are plotted as straight lines as predicted by the Summerfield Equation. Each point represents the arithmetic mean of at least 3 runs; all data lie within  $\pm$  4 per cent of the arithmetic mean points. The intercept, "a", and slope, "b", calculated on the basis of Eq. 2 for each combination of  $\delta_0$  and  $\delta_f$  are tabulated in Table 1 (as are values of "b" and "a" for the propellants of Ref. 4 and Ref. 5).

Contrary to the prediction of the Summerfield Equation, the values of "a" obtained from the loose-granule burner data at constant mixture ratio are not constant. This deviation is also typical of propellant data (as pointed out earlier) and since both deviate in this way the analogy between loose-granule burner and composite propellant behavior appears to be strengthened. Further support of the analogy is provided by the fact that values of "a" for the burners and for propellants are quite close. (Differences in system chemistry and mixture ratio prevent exact comparison on this point.) Also, there is close agreement between the values of "b" from burner data and those from propellant data presented in Table I. (Again differences in system chemistry and mixture ratio

prevent exact comparison.)

To allow further comparisons between loose-granule burners and propellants, Fig. 15 shows b vs  $\delta$  data for loose-granule burners; in one series of tests  $~\delta_{_{\mathrm{f}}}$  was held constant while  $\delta$  was varied and in the other  $\delta$ held constant and  $\delta_f$  varied. While b vs  $\delta_o$ for propellants have been found to be linear within certain limits of parameter variation, these loose-granule burner data are not linear within the limits of parameter variation tested. The granularities, both  $\delta_{_{
m C}}$  and  $\delta_{_{
m f}}$  , of the loose-granule burners employed in obtaining the data displayed in Fig. 15 are much greater than the oxidant granularity of propellants that exhibit linear characteristics in this plane (See Fig. 3). Hence it is reasonable to presume that the loose-granule burner data obtained in this investigation are analogous to propellant data obtained in the "turbulent burning regime" (defined in Ref. 5) wherein propellants do not display a linear proportionality between "b" and the granularity, . Note that both Figures 3 and 15 show "b"  $\cong$  40 for  $\delta_{\rm o}$  > 150  $\mu$  .

# ii Comparisons via the P/r vs 8 plane

In addition to consideration of loose-granule burner data plotted in the P/r vs  $P^{2/3}$  planes and the values of "a" and "b" determined by such plots, the measured

deflagration characteristics of the loose-granule burners may be compared with those of propellants via the P/r vs  $m{\delta}_0$  and P/r vs  $m{\delta}_f$  planes. Data taken with various oxidant and fuel granule combinations are plotted in the P/r vs  $m{\delta}_0$  plane (Fig. 16) and P/r vs  $m{\delta}_f$  plane (Fig. 17) for constant  $m{\delta}_f$  and  $m{\delta}_0$  respectively, with P as a parameter. The data for varying  $m{\delta}_0$  did not correlate to straight lines in the P/r -  $m{\delta}_0$  plane for either of the two fuel diameters used. However, data plotted in the P/r vs  $m{\delta}_f$  plane (Fig. 17) did indicate straight line correlations within a limited pressure range (see p. 37).

One reason for this deviation from the expected result might be that the dispersion of fuel granules throughout the more populous oxidant crystals was not uniform from run-to-run. Assuming a uniform distribution of fuel granules, the fuel spacing is independent of  $\delta_0$  at constant mixture ratio. And since the dependence of r on  $\delta_0$  is weak (Fig. 16), it is concluded that if the loose-granule burner r has a dependence on "granularity", it must be as a result of the spacing between fuel granules or  $\delta_f$ . Fig. 17 indicates that  $\delta_f$  is a sensitive parameter in this regard.

This, at first, might appear to indicate a dissimilarity in the granularity dependence of burning rates in propellants and in the loose-granule burner. For a propellant of constant mixture ratio, fuel "granularity" is a function of  $\boldsymbol{\delta}_{0}$  only (See Fig. 1) and, therefore, when  $\boldsymbol{\delta}_{0}$  is varied  $\boldsymbol{\delta}_{f}$  is also varied. In reporting propellant data the parameter usually used is  $\boldsymbol{\delta}_{0}$ , a more readily measured scale of granularity; however, the possibility exists that it is not the oxidant granularity but the fuel "granularity" which determines the scale of granularity for the combustion mechanism of composite solid propellants. And as discussed in the <u>BACKGROUND AND INTRODUCTION</u>, this question cannot be resolved by experiments performed with composite solid propellants (See Fig. 1).

Since the deflagration characteristics of the loose-granule burner has been demonstrated to be so similar to those of composite propellants in other regards, it seems reasonable to assume that it will be useful in illuminating the true source of composite propellant deflagration rate dependence on propellant granularity. But sufficient information is not at hand to do so at this time.

From the foregoing views of loose-granule and propellant data in both the P/r vs  $P^{2/3}$  plane as well as in the P/r vs  $\mathbf{\delta}_{\mathrm{f}}$  and  $\mathbf{\delta}_{\mathrm{o}}$  planes, it is significant to compare the common aspect of both views. Slopes of the loose-granule burner straight line correlations in the

P/r - P<sup>2/3</sup> and P/r - \$\mathbb{6}\_f\$ planes were computed and from these slopes, values of b' were determined via Eq. 3. These values as well as those of the propellants of Refs. 4 and 5 are tabulated in Tables 1 and 2. It is apparent that, just as values of b' from propellant data are different depending on whether they are determined from P/r vs P<sup>2/3</sup> or P/r vs \$\mathbb{6}\_O\$ correlations, so are values of b' from the loose-granule burner data. It is concluded, that, while burner and propellant behavior are analogous, Eq. 3 misrepresents both in much the same manner and, therefore, strengthens the analog.

It should be noted, finally, that the distribution of AP particle sizes employed in manufacture of the propellants of Ref. 4 encompassed a much broader size range than that employed in the manufacture of the propellants of Ref. 5. As the assumptions leading to Eq. 3 are based on a  $\mathbf{6}_{0}$  of a single size, the data obtained from the Ref. 5 propellants should be represented by the present scheme much better than those of Ref. 4 propellants. This, in fact, appears to be true, i.e. the values of b' yielded by the Ref. 4 propellants were much more scattered than those yielded by the Ref. 5 propellants. This same condition is expected to hold with the loose-granule burner, i.e. the narrower the particle size distributions used, the less dispersed the apparent granularity dependence of

loose-granule burning rates as characterized by the parameter b'. In this light it is noteworthy that values of b' determined from the loose-granule burner data of this study fall between 0.05 and 0.10 and, thereby, display less dispersion than the values of 0.11 to 0.37 for the Ref. 5 propellant. This is further evidence for the analog.

In summary, the validity of the loose-granule burner technique as an analog to the composite propellant deflagration mechanism has been established. It is expected that the burner will become a primary tool for evaluation of the various proposed models of the propellant deflagration process. As fabrication of this type of burner does not require propellant casting, as does preparation of composite propellant samples, the burners are less expensive and safer to prepare, and, therefore, are quite suitable for experimentation in typical university laboratories.

## E. Visual Observations of the Burner Deflagration

## i The AP porous-plug burner

The AP porous-plug burner with a of 297 - 500 pm has been operated at conditions from fuel-lean to fuel-rich and the deflagration process photographed.

For  $\dot{m}_f/\dot{m}_O$  below 0.045 and at atmospheric pressure

photographic study of the porous-plug burner combustion has revealed the following: the flame is visible (pale orange) and apparently laminar within the tube; the regressing surface is slightly concave with a film of unburned material adhering to the wall of the burner and extending approximately 1/4" above the surface. This region corresponds to the 15 psia curve of Fig. 8 for  $m_f/m_o$  less than 0.045.

For  $m_f/m_O$  above 0.070 an orange-blue flame appears at the top of the tube, presumably supported by combustion of unburned fuel with oxygen in the surrounding environment. Therefore, it is concluded that the combustion within the tube is fuel-rich. This region corresponds to values of  $\mathring{\text{m}}_{\text{f}}/\mathring{\text{m}}_{\text{O}}$  greater than 0.070 for the 15 psia curve of Fig. 8. The regressing surface is slightly convex and no material adheres to the walls. The flame in the tube, the primary flame, is pale orange and appears to be somewhat turbulent at values of  $\dot{m}_f/\dot{m}_O$  greater than For operation with  $m_f/m_O$  above 0.085, individual particles can be observed leaving the surface and subsequently burning downstream (Fig. 9). For other AP porous-plug burners with  $\delta_{\odot}$  of smaller size, individual particles less than 210 m diameter could not be seen leaving the surface (apparently because of their small size); however, clusters of particles were elutriated by

the throughput gas and could be observed.

There is a region between the region of the concave surface and the convex surface where a transition occurs. In this region,  $0.045 < \dot{m}_f/\dot{m}_O < 0.070$ , the regressing surface is flat with little material extending up the walls. And the primary flame is pale orange and laminar.

### ii The loose-granule burner

Ideally, fuel and oxidizer particles are distributed randomly throughout the loose-granule burner without local clustering of either specie. This desirable randomness was not, however, achieved.

It was observed prior to packing of the PS - AP mixture into the burner tube that, for PS particles of less than 200  $\mu$ , clusters of PS formed in the mixture. Further, in mixtures of large PS particles (500-420  $\mu$ ) and smaller AP particles (177-149  $\mu$ ), it was observed that AP particles adhered to the PS particles (Figure 18). Microscopic observations of small samples of the particulate mixtures indicated mutual attraction among particles, apparently a consequence of the accumulation of electrostatic charge by the PS.

It was further observed that some of the mixed AP and PS granules adhered to the walls of the container in

which they were stored before being packed into burner tubes. The relative proportions of AP and PS which adhered were not determined and so it is possible that a change from the nominal fuel-oxidizer mixture ratio occurred when the mixture was transferred to the burner tube.

These observations relative to possible non-randomness of mixing and changes in mixture ratio were not considered in data reduction and interpretation; in the first case, because the specie clustering was not observed for all mixtures and is believed to be of small effect in most instances, and, i.. the second case, because the proportion of granules adhering to the container walls was small.

The regressing surface of the loose-granule burner was observed to be flat in tests at atmospheric pressure with no flame penetration below the surface. At combustion pressures above 37 psia, however, visual and photographic observation indicated flame penetration below the surface and unburned solid material leaving the surface. The onset of such interstitial flame penetration with increasing pressure was observed to correspond with the beginning of the "transition regime" of Fig. 12. Interstitial flame propagation is responsible for the increased scatter of the highest pressure data of Fig. 17.

Deposition of solid carbon on the burner walls during combustion occurred in some tests. The 82% AP - 18% PS mix-

ture deposited carbon while the 90% AP - 10% PS and 86% AP - 14% PS mixtures did not. While these observations may bear on the question of combustion stoichiometry, the connection is not clear and was not investigated further.

#### SECTION IV

#### SUMMARY

The ammonium perchlorate composite propellant deflagration process is so complex that, for the present, tractable mathematical analysis of the complete problem is impossible. In order to produce solutions sweeping, heuristic assumptions have been made which have resulted in simplistic models of the actual process; these solutions normally take the form of r(P,  $\delta_{o}$ ,  $T_{f}$ , etc.). The workers in the Combustion Laboratory have concluded that the validity of even these simplistic models can not be established merely by comparing propellant data with such predictions (Ref. 6) - - the models themselves must be tested, but unfortunately it is impossible to perform the required tests with actual composite propellants due to their peculiar physical structure. Other means must be found. In the Combustion Laboratory two types of burners have been developed for this purpose. Results obtained with these burners have helped clarify the nature of the pivotal boundary condition for ammonium perchlorate deflagration (Ref. 6).

Reported herein are the results of recent experiments with these burners. They indicate that:

- (1) The loose-granule burner technique represents an excellent, if not perfect, experimental analog of the propellant deflagration process. In the pressure range where the Summerfield scheme can be employed to characterize propellant combustion characteristics (roughly, below 50 psia) comparison of propellant characteristics with suitable burner characteristics revealed that: (a) both have about the same surface regression rate, (b) values of the parameters "a" and "b" are remarkably similar (considering the inherent differences, chemical and otherwise, of the systems) and show similar trends with variation of experimental parameters.
- (2) In light of (1) and the demonstrated greater sensitivity of burner  ${\bf r}$  to  $\delta_{\bf f}$  than to  $\delta_{\bf o}$ , the geometry of thermoplastic fuel-binder pockets might have an unexpected-ly strong influence on the propellant deflagration process -- although careful study of the influence of burner  $\delta_{\bf o}$  and  $\delta_{\bf f}$  variation on system geometry is required before a definite conclusion can be drawn.
- (3) The role of the thermoplastic fuel-binder in the deflagration process cannot realistically be described simply in terms of its overall chemical energy release -- the details of the energy release rate distribution pattern through the flame zone have an influence on the deflagration

rate, at least insofar as PMM and PS are concerned. Although this was indicated as a result of previous work (Ref. 6), the subject results add significant support to this view.

(4) The bonding technique used for fabricating porous-plug burners has a strong influence on burner deflagration characteristics and this weakens the inferences drawn from interpretation of them. Bonding of burners with a saturated water - AP solution while not introducing foreign cements into the burner results in an analog technique limit close to the mixture ratio resulting in maximum  $\Gamma$ . This fact prevents extracting unambiguously the value of the stoichiometrically correct mixture ratio from porous-plug regression rate data. Further, bonding with PMM cement results in a dependence of the mixture ratio for maximum r on the PMM concentration of the bonding solution (although it permits data to be gathered for mixture ratios far beyond the limit of the AP-water-solution-bonded burners). This dependence on cement composition also prevents unambiguous interpretation of porousplug data in terms of the stoichiometrically correct mixture ratio. Thus, it appears that this ratio cannot be determined by measuring only the regression rates of porousplug burners. The work of Ref. 10 should be re-examined in

light of this result.

While limitations of porous-plug and loose-granule burners were determined during the subject program, there is, nonetheless, no indication that the burner analog technique is not the best available means for testing the validity of proposed models of the deflagration of composite solid propellants containing ammonium perchlorate.

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#### APPENDIX A

### Burner Fabrication

## i Burner tube description

Two types of burner tubes were employed. To permit visual and photographic observation of the combustion process, heat resistant glass ("Pyrex") tubes (0.51" O.D. X 0.47" wall) in lengths of 6" or 8" were used. For regression rate measurement stainless steel tubes (0.50" O.D. X 0.049" wall) in lengths of 6" or 8" were used. At each of 3 equidistant stations along the tube length, 2 radial holes were located and drilled, using a Number 60 drill, through opposite sides of the steel tube. A timing wire, which consisted of a 6" length of fuse wire (Bussman 2 amp), was then threaded through the tube at each station. At the six points where the wires pierced the tube wall the wires were coated with ceramic cement ("Sauereisen") to insure rigid placement and to insulate the wires electrically from the tube. In both types of tubes, paper wadding was used at the lower end of the tubes to support the loose granules within the tubes.

## ii Bonding of the porous-plug burners

Several techniques for bonding the loose AP granules to form a strong porous-plug have been employed in this laboratory. Two previous methods have been reported (Ref. 7 and 8) but subsequent investigation revealed that the reproducibility of regression rate data from burners made using these techniques was unsatisfactory.

The first bonding method (Ref. 7) involved subjecting the tubes, fully loaded with AP granules, to a quiescent atmosphere of moist air for approximately six hours and subsequent drying in an oven maintained at 100°F. However, by drilling through or burning away the end of the plug, it was found that the bonding extended only to a depth of approximately 1" in some cases, so that the underlying AP remained in its original unbonded condition. This indicated that the moist quiescent atmosphere was incapable of producing a uniformly bonded condition throughout the length of a 6" or 8" plug, a prerequisite for reproducibility. This technique was abandoned.

The second technique (Ref. 8) depended upon the fact that water is a solvent for AP whereas ether is not. A 25% (by volume) solution of water in ether was

permitted to gravitate through a tube containing loose AP granules. It was hoped that this water content would be sufficient to form a point of contact bond between granules but insufficient to dissolve the AP particles appreciably. Several deficiencies were found in this technique. Examination of a 25% "solution" of water in ether revealed that almost none of the water remained in solution with the ether but had settled out. Thus, granules subjected to this mixture would be immersed either in water, causing partial dissolution of AP and hence greatly reducing the size of the particles in the tube, or in ether, producing no bond at all.

These drawbacks were eliminated with the present bonding technique that finally evolved. A tube filled with AP granules is compacted by tapping 75 - 100 times and the resulting empty space at the top filled with more AP. The bonding solution is prepared by dissolving AP in water at 150°F until a saturated solution is obtained, and then cooling to room temperature before use. A vacuum is drawn at the bottom of the tube as the bonding solution is poured through the packed granules - 1.5cc of solution per inch of packed plug.

After the solution has wet the entire length, suction is continued for 3 minutes to initiate drying. The tube is placed in a constant temperature oven (at 150°F.) for 6 hours, cooled to room temperature, and dry air is then passed through for one additional hour.

The porous-plugs bonded by the above system did not exhibit those properties which invalidated the previous techniques. The bonding solution insures a minimum reduction of the selected AP granule size. Examination of the plug via the methods used on the previous systems proved that the porous-plug was bonded throughout its entire length.

The porous fuel plug is produced in a similar manner to that of the oxidant plug. The major differences are that methyl ethyl ketone is used as the bonding solution and that no heat is used in drying the tubes.

#### iii Loose-granule burner

Fuel (PS) and oxidant (AP) were blended mechanically (Appendix C) or manually in varying weight ratios in 300 gram quantities. There was no noticeable difference in the physical appearance of the mixture (or data produced) with either blending technique. Tubes

were then filled with the mixture, tapped 75 - 100 times, and the resultant void refilled. Tapping is an important step since uncompacted tubes have exhibited up to 15% faster burning rates than compacted ones.

### iv Igniter

The igniter charge consisted of a mixture of 14% ps - 86% AP by weight. A 1½" length of #30 gauge nichrome wire was used as the heat source. A length of paper (drafting) tape was wrapped around the top of each burner tube forming a ½" extension of the tube wall. The tape was pierced on opposite sides and the wire threaded through, leaving ½" projecting on the outside of the tape for the electrical connections. The charge was then poured into this extension, covering the wire. With porous-plug burners, three drops of acetone were added to the top surface of the igniter charge in order to bond the granules and thereby prevent elutriation by the throughput gas. With the loose-granule burners no bonding was required.

#### APPENDIX B

### Pressure Housings

During operation of the original pressure housing (Ref. 7), the combustion products accumulate in the housing and prevent visual observation of the regressing surface through the windows. This condition necessitated the construction of a new pressure housing from which these combustion gases could be continuously exhausted.

The new housing was a Penberthy high pressure boiler sight glass (Penberthy Co., Prophetstown, Ill., Model No. WT-13). The installation of a critical flow orifice in the exhaust line permitted the establishment of the desired equilibrium chamber pressure during operation. The bottom of the chamber was fitted with a 2½ inch pipe union. The female end was connected to the housing and the male end closed by a steel cap. The cap was constructed to accept a burner tube holder, pressure tap, electrical tap and nitrogen gas supply line. The pressure gages, ignition source and methane flow system were used interchangeably with both the original and the modified housings. Figs. 5 and 6 are schematic of the original and the modified housings, respectively, with their support equipment.

#### APPENDIX C

## Timing Systems

During the course of the subject program, two timing systems were used.

Initially, a system was employed incorporating single pole, double throw relays (Potter and Brumfield Co., Model PW5DS - 6vdc.) with automatic reset. Fig. 19 is a wiring diagram of this system. This circuit, however, proved inadequate since a short circuit could be caused by a melting timing wire making contact with either the tube or another wire.

The possibility of short circuits was eliminated by an improved system which used stacked, double pole, double throw relays (Kurman Electric Co., Model KX1H2E, 6 vdc.) providing for manual resetting. Fig. 20 is a wiring diagram of this system.

Two pairs of timing clocks (Standard Electric Time Co., Model S-13H-10 and Model S-1) were used interchange-ably in the two timing systems.

#### APPENDIX D

### Materials and Classification

## i Ammonium Perchlorate

The ammonium perchlorate used in this investigation was purchased from the American Potash and Chemical Company, Los Angeles, California, and did not contain an anticaking agent. Particle size range was specified and various weights of material were ordered at different times. Table 3 displays a sample analysis of three separate orders.

## ii Polystyrene

The polystyrene used in this study was purchased from the Sinclair-Koppers Company, Plastics Division, Monaca, Pennsylvania, under the trade name of Dylene 8x. The material was ordered in two size ranges, 30 fines, (500 to 200 $\mu$ ), and 60 fines, (250 to 50 $\mu$ ). Rounded beads were specified.

## iii Polymethylmethacrylate

The polymethylmethacrylate used in this investigation was purchased from Rohm and Haas Co., Philadelphia, Pa., under the trade name VM - 100 Plexiglas. The material was ordered as molding powder (colorless

pellets) approximately 1/8" in diameter. These pellets were ground by hand with mortar and pestle to obtain small sizes.

## iv Classification of Granules

A series of screens (Tyler) were employed to separate the "as received" granular materials into fractions having a specific range of granule diameter. A Ro-Tap sieve shaker (Tyler Company, Cleveland, Ohio) was used to vibrate the screens and thus move the granules through them. This granule size classification method was successful for AP granule diameters greater than 74 \( \mu \). Below 74 \( \mu \) the screen holes tend to clog making further classification extremely difficult. Unfortunately, with the polystyrene granules less than 210 \( \mu \) in size, accumulation of electrostatic charge by the granules caused them to adhere to the screens. Hence, granules less than 210 \( \mu \) could not accurately be classified by this method.

BURNER AND COMPOSITE SOLID PROPELLANTS: PRESSURE VARIED DEFLAGRATION CHARACTERISTICS OF THE LOOSE-GRANULE TABLE I.

	FUEL	AP	w T	ç	ro	Q.	<b>1</b>
		(wts)	Ê	Ĵ.	(1b/in; (in./sec)	(lb/in: / (in./sec)	(lb/in; )? (in./sec) (μ)
		98	420-350	420-500	165	41	0.11
AP	POLY-	98	297-250	250-210	120	33	0.12
1,0008-		82	500-420	500-420	240	. 92	0.05
1 2000	STYRENE	:	=	350-297	195	39	80.0
GRANULE		:	=	297-250	190	36	80.0
RITRNER			=	210-177	195	34	0.07
		:	2	177-149	195	39	0.05
		=	420-350	420-500	235	25	90.0
		=	350-297	2	200	28	60.0
		=	297-250	=	200	56	0.10
		:	350-297	350-297	220	27	80.0
		=	z	297-250	165	33	0.10
		06	420-350	500-420	135	51	0.13
AP		65	f ( <b>&amp;</b> _0)	270-190	+06T	45+	0.20
COMPOSITE	POLY-	=	=	230-130	130+	44+	0.25
	CIII BINE	=	8	180-100	130+	40+	0.29
SOLID	2012	=	8	140- 60	150+	36+	0.37
PROPELLANT	(Ref.5)	E	#	120- 40	210+	24+	, 0,31

TABLE I. CONTINUED

FUEL AP $(\text{wt}8)$ $(\boldsymbol{\mu})$ $(\boldsymbol{\mu})$ $(\boldsymbol{\mu})$ $(\text{in./sec})$ $(\text{in./sec})$ $(\text{in./sec})$ $(\text{in./sec})$ $(\boldsymbol{\mu})$				80 " 16 250* 17* 1.06+	
FUEL	POLY- ESTER- STYRENE (Ref.4)				
	AP COMPGSITE SOLID PROPELLANTS				

EXTRACTED FROM DATA PRESENTED

AS REPORTED

DEFLAGRATION CHARACTERISTICS OF THE LOOSE-GRANULE BURNER AND A COMPOSITE PROPELLANT: PRESSURE CONSTANT TABLE 2.

THE TOTAL CONTROL OF THE PROPERTY OF THE PROPE

	FUEL	AP (wt&)	& (* #)	જ્રું	P (psia)	(1b/in.2)	(psia) (1b/in.2) (1b/in.2)
AP		06	varied	297-210	atm	270	0.06
LOOSE-	POLY-	98	varied	210-297	=	250	0.07
GRANULE	STYRENE	82	varied	420-500	16	300	80.0
BURNER		82	varied	=	23	340	0.07
AP	POLY-	65	£(8°)	varied	15	300+	0.11+
COMPOSITE	SULFIDE	=	=	=	30	360+	0.12+
SOLID	(Ref.5)	<b>=</b>	z	:	50	430+	0.13+
PROPELLANT		•					

+ EXTRACTED FROM DATA PRESENTED

TABLE 3: TYPICAL AMMONIUM PERCHLORATE ANALYSIS

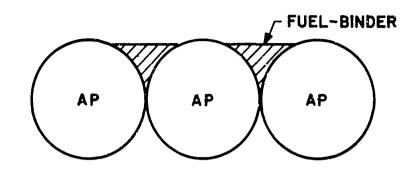
Supplier: American Potash and Chemical Corporation,

Los Angeles, California

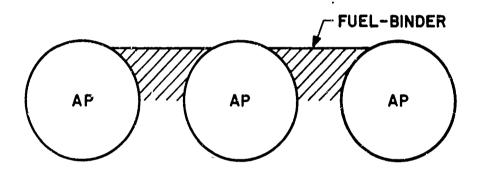
# Analysis of Material

	Batch #1	Batch #2	Batch #3
NH <sub>4</sub> ClO <sub>4</sub> , %	99.7	99.7	99.7
NH <sub>4</sub> Cl, %	0.03	0.013	0.018
NaClO <sub>3</sub> , %	0.006	0.008	0.006
Moisture total, %	0.02	0.019	0.082
Sulfated Ash, %	0.09	0.06	0.08
(NH) 2 SO4, %	Trace	Trace	Trace
Insoluble acid, %	0.003	0.003	0.006
Metal Oxides, % (Non alkali)	0.009	0.004	0.002
Na Bromate, %	0.001	0.0008	0.001
pH Value	5.4	,5.0	4.7
Particle Size Dist	ributions		
۶ مر420 ک			45.8
۶ , بر350 <			90.7
>297 <sub>M</sub> , 8	2.8	6.6	99.1
۶ , بر <sup>210</sup> ک		42.7	
۶ بر <sup>49</sup> و ۱	79.6	73.9	
>105µ, %	90.8	91.0	
> 74 <sub>M</sub> , 8	98.8	98.6	
> 44m, 8	100.0		

# NEAR STOICHIOMETRICALLY CORRECT MIXTURE



# **FUEL-RICH MIXTURE**



# MIXTURE HELD CONSTANT

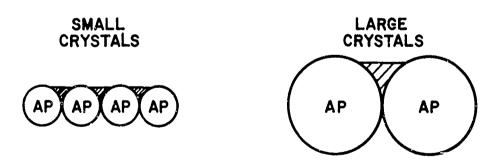
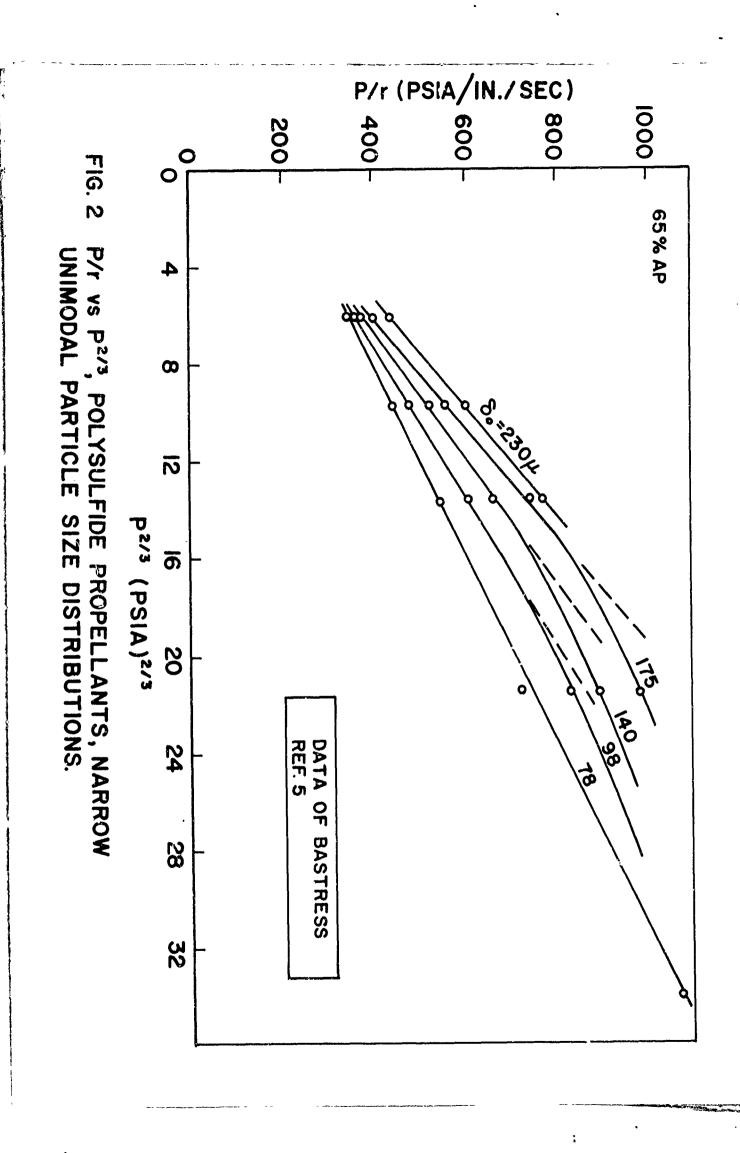
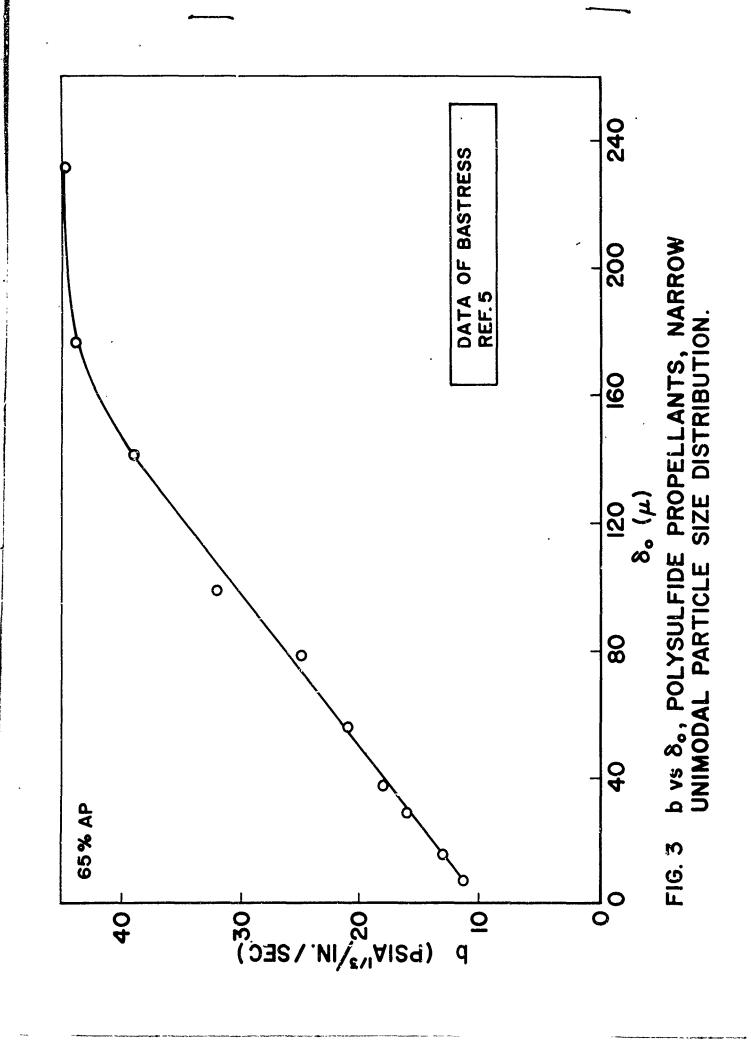


FIG. I VARIATION OF FUEL-BINDER GRANU-LARITY WITH AP CRYSTAL SIZE AND MIXTURE RATIO.





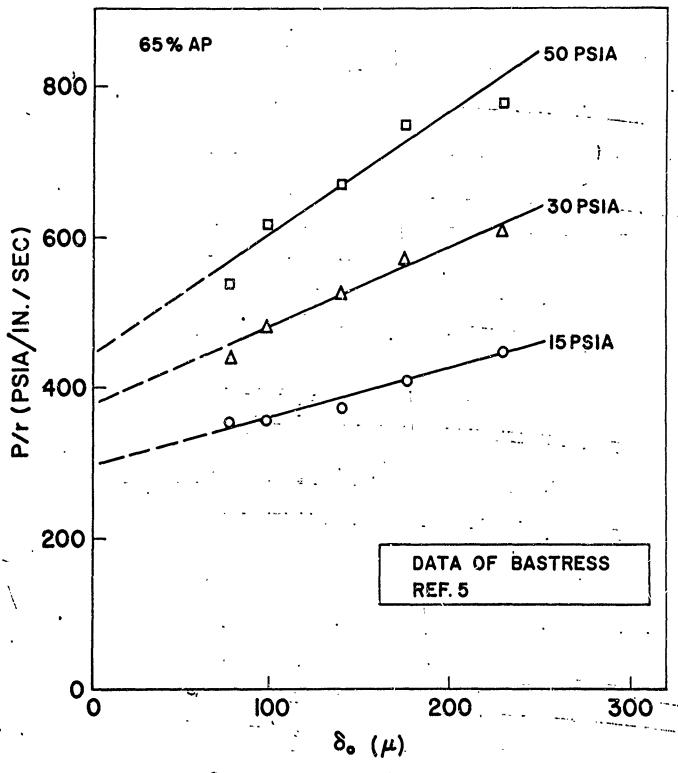
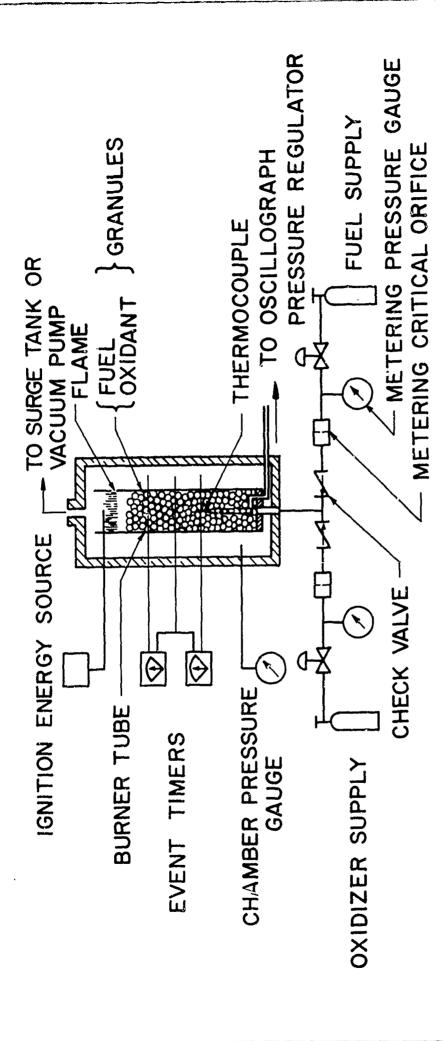


FIG.4 P/r vs 8. AT VARIOUS PRESSURES FOR POLYSULFIDE PROPELLANT



SCHEMATIC OF COMPOSITE SOLID PROPELLANT DEFLAGRATION SIMULATOR (POROUS-PLUG BURNER DEPICTED) F16.5

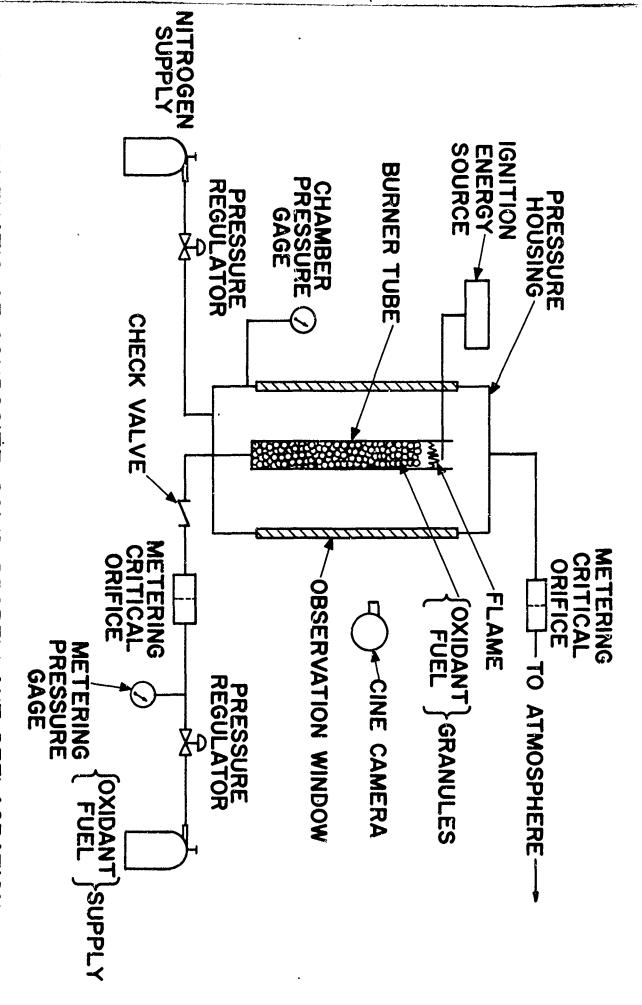
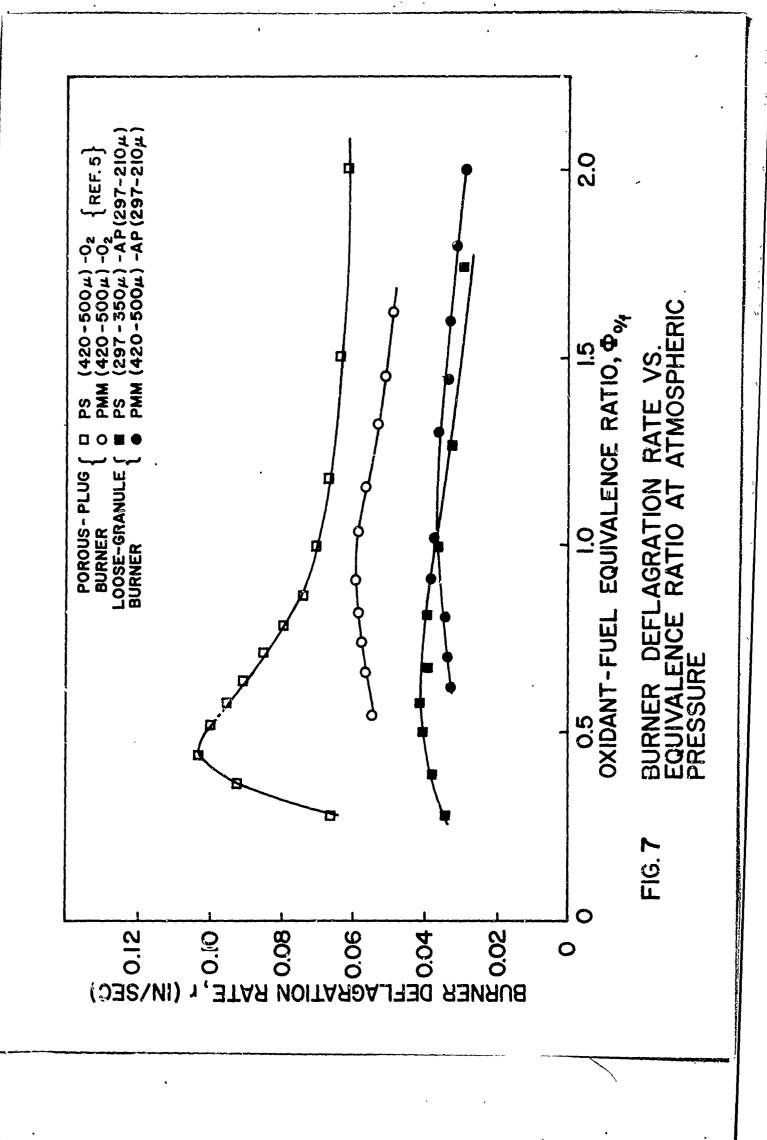


FIG. 6 SCHEMATIC OF COMPOSITE SIMULATOR (POROUS-PLUG BURNER DEPICTED): MODIFIED FOR OPTICAL STUDIES. SOLID PROPELLANT DEFLAGRATION

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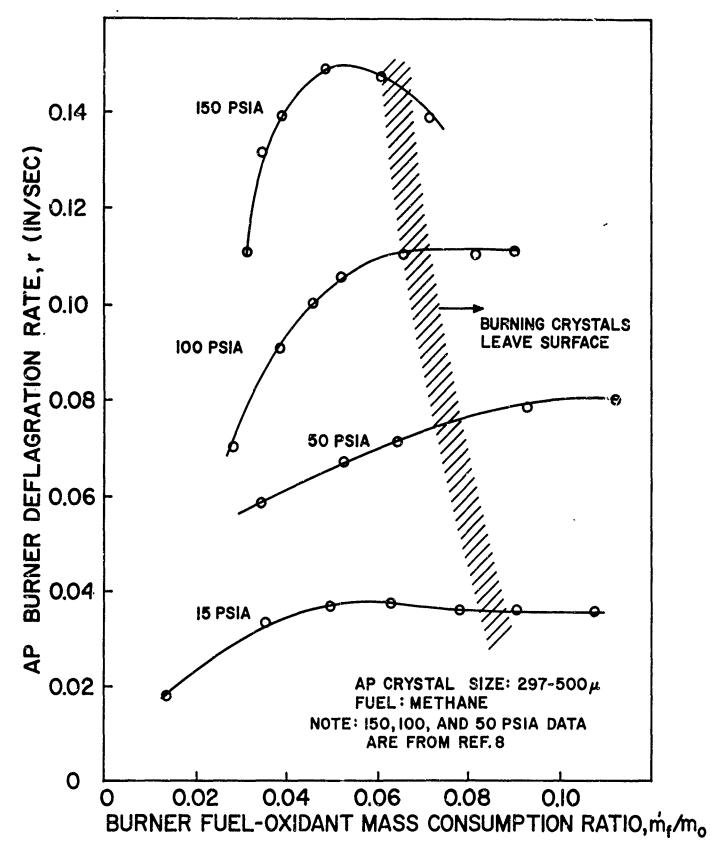
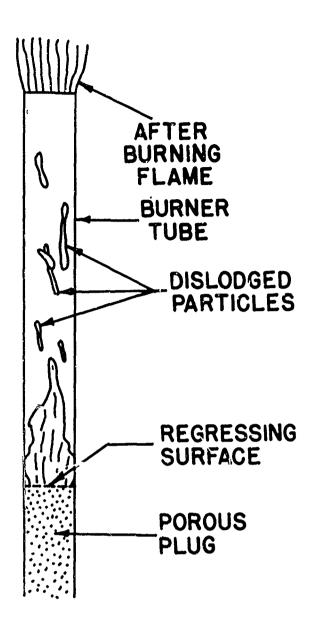


FIG. 8 AMMONIUM PERCHLORATE BURNER DEFLAGRATION RATE VS. BURNER FUEL-OXIDANT MASS CONSUMPTION RATIO AT VARIOUS PRESSURE LEVELS.





 $\dot{m}_f/\dot{m}_o$ : 0.09 PRESSURE : 15 psia

AP CRYSTAL SIZE:  $500-297\mu$ 

FIG. 9 PHOTOGRAPH OF BURNING AP PARTICLES LEAVING POROUS-PLUG SURFACE.

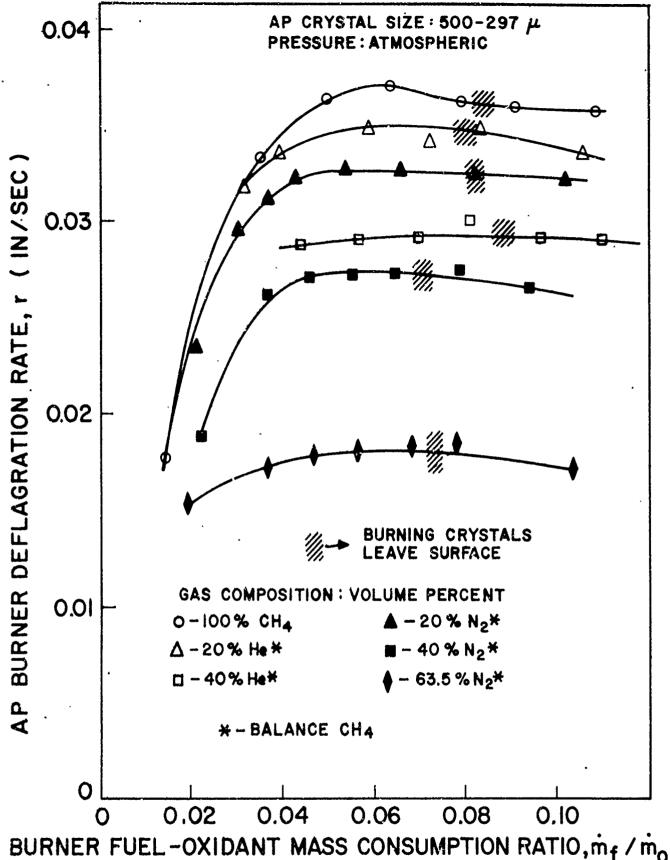


FIG.IO AMMONIUM PERCHLORATE BURNER DEFLAGRATION
RATE vs BURNER FUEL-OXIDANT MASS
CONSUMPTION RATIO FOR VARIOUS FUEL DILUTIONS

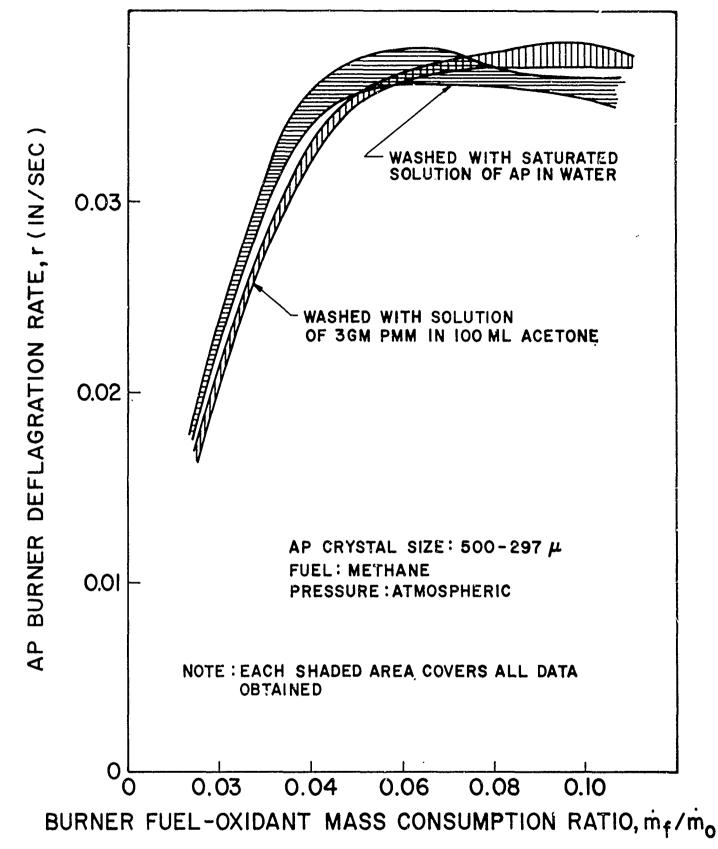


FIG.II COMBUSTION CHARACTERISTICS OF AP POROUS — PLUG BURNERS FABRICATED BY TWO DIFFERENT TECHNIQUES.

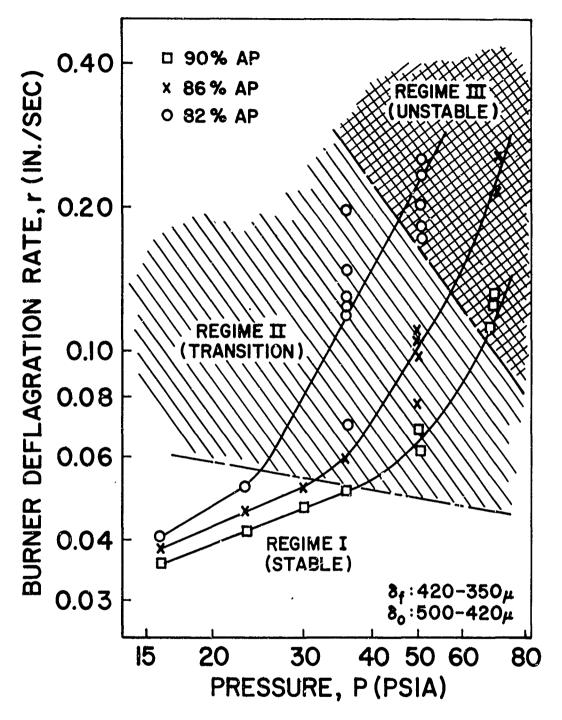


FIG. 12 LOG P vs LOG r AT VARIOUS MIXTURE RATIOS FOR THE LOOSE-GRANULE BURNER.

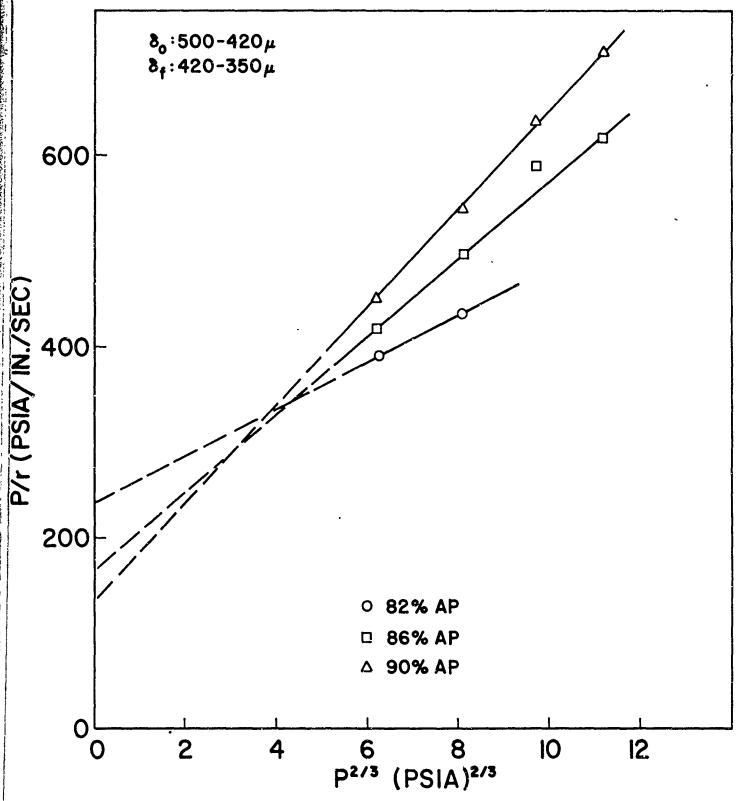


FIG. 13 P/r vs P2/3 AT VARIOUS MIXTURE RATIOS FOR LOOSE-GRANULE BURNER.

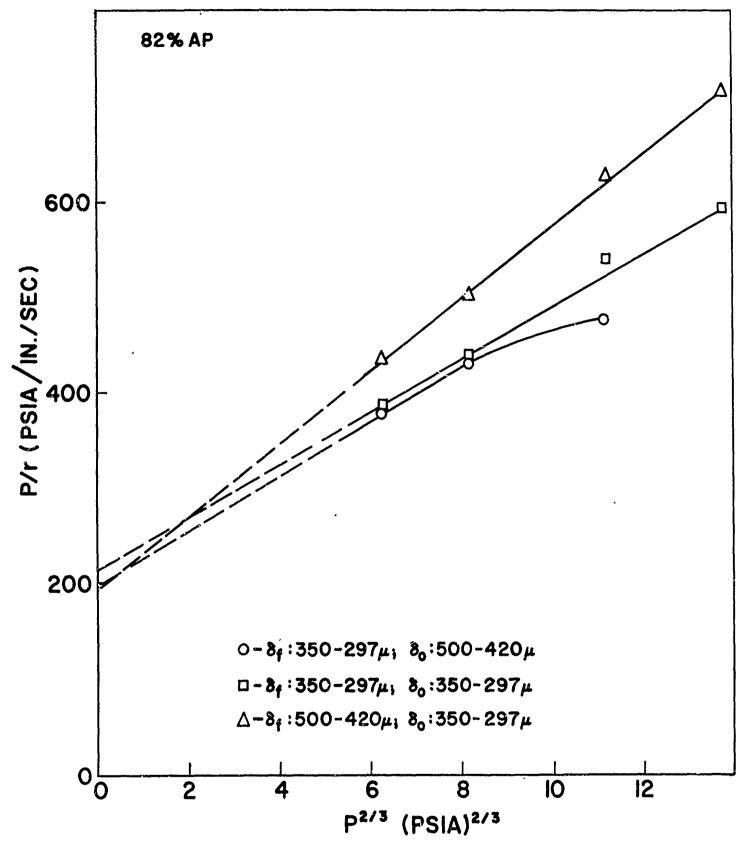


FIG. 14 P/r vs P<sup>2/3</sup> AT VARIOUS GRANULARITIES FOR LOOSE-GRANULE BURNER.

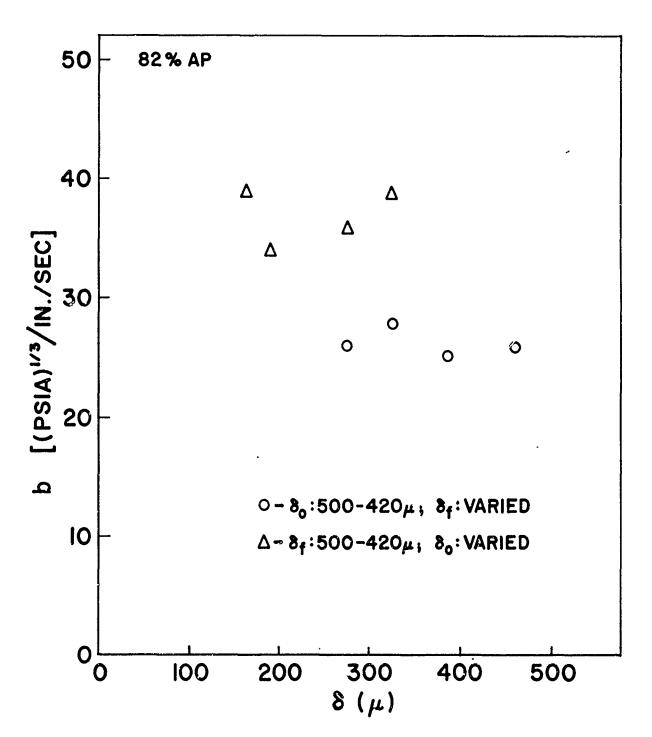
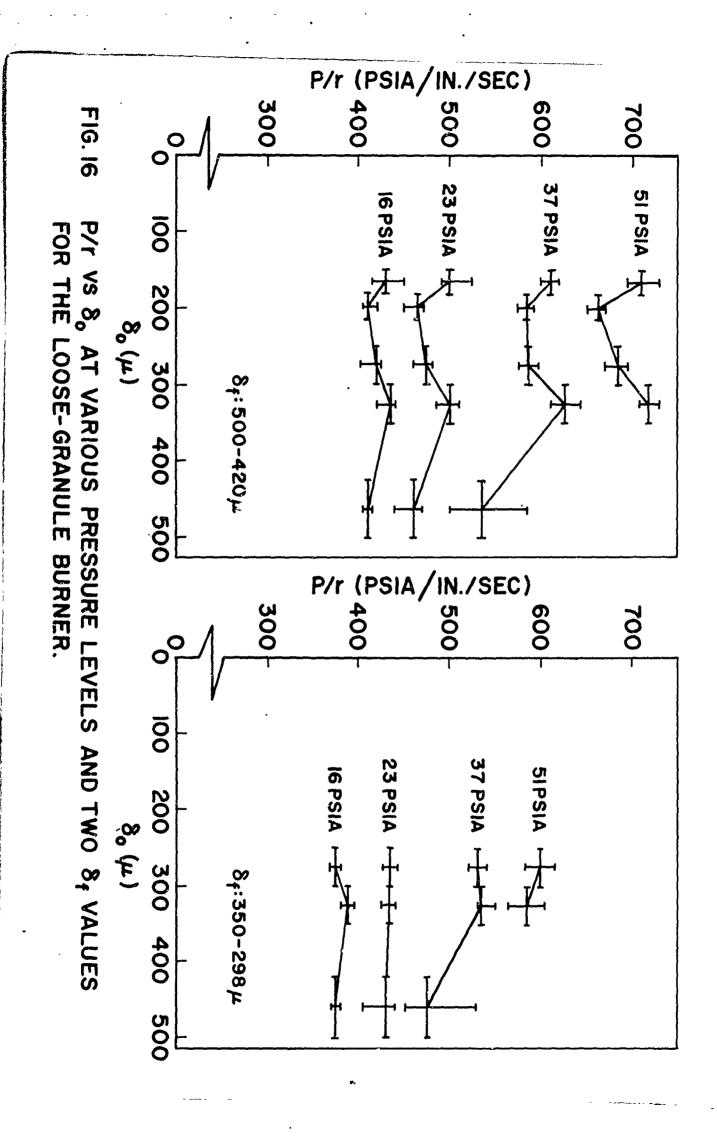


FIG. 15 b vs 8 FOR LOOSE-GRANULE BURNER.



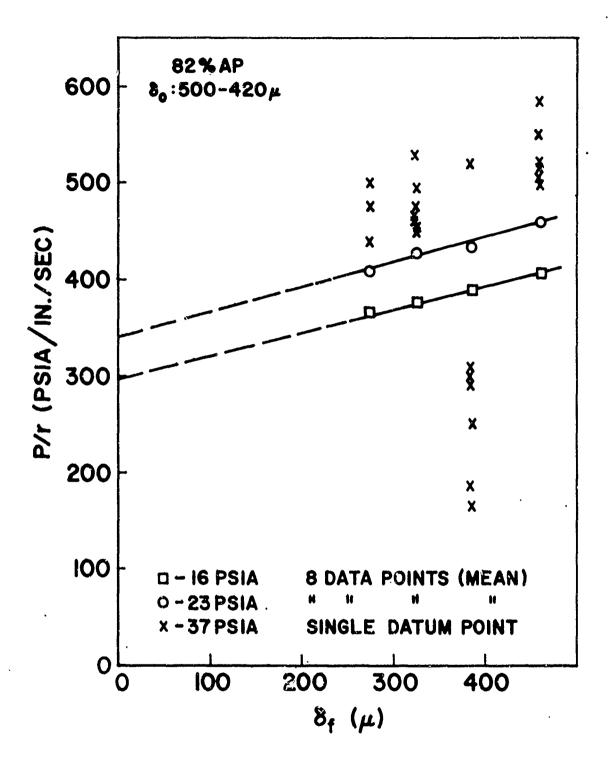
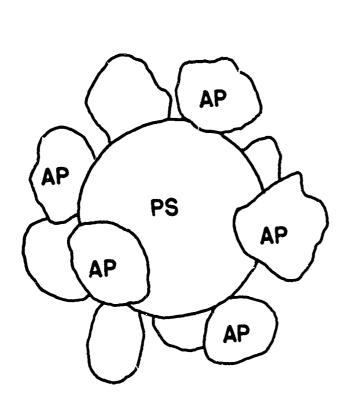


FIG. 17 P/r vs & AT VARIOUS PRESSURE LEVELS FOR THE LOOSE-GRANULE BURNER.





82% AP  $\delta_0$ : 177 - 149  $\mu$ 

 $\delta_{\rm f}$ :500-420 $\mu$ 

FIG. 18 ILLUSTRATION OF SMALL AP GRANULES ADHERING TO A LARGE PS GRANULE.

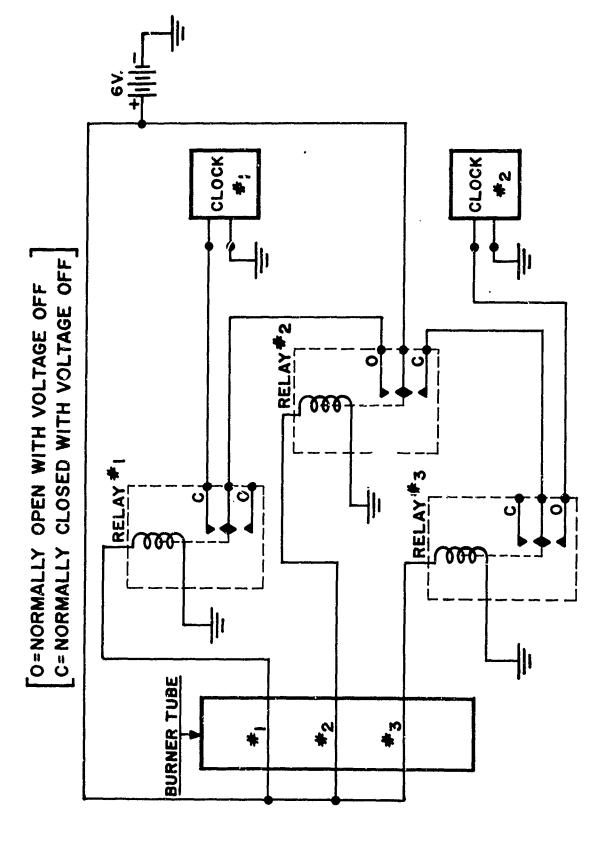


FIG. 19 TIMER SYSTEM CIRCUIT SCHEMATIC (AUTOMATIC RESET)

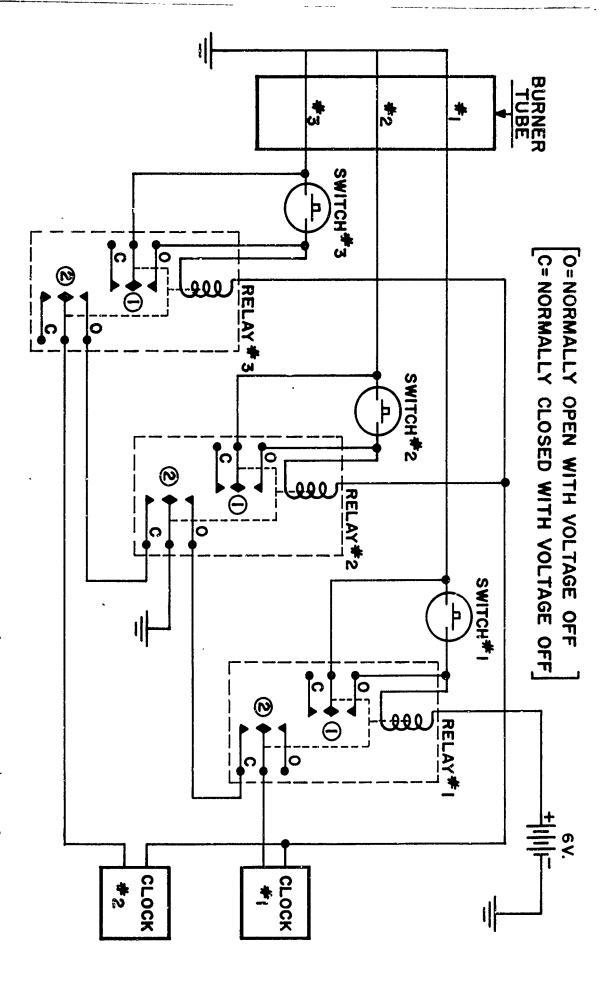


FIG. 20 TIMER SYSTEM CIRCUIT SCHEMATIC (MANUAL RESET)

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13. ABSTRACT Two types of experimental, an	alogue burners	have-be	WELE enideveloped and		
employed for the purpose of evaluating					
deflagration. One type, the "porous-					
ammonium perchlorate (AP) bed burning					

porous fuel beds may be burned with oxidant gas throughputs. The other type the "loose-granule" burner, involves mixtures of solid AP and fuel granules burned without a gas throughput. These analog burners allow evaluation of parametric influences including fuel and oxidant initial physical phase, system chemistry, and oxidant and fuel granularity scales. Such evaluation based on combustion of propellants themselves is prevented by their structural peculiarities. Experimental results from the two analog burners suggest that deflagration rates depend on the spatial distribution of energy release rate and not on overall energy release alone. Loose-granule burner deflagration is shown to be an excellent, if not perfect, analog of actual propellant deflagration; experiments with the burner show that fuel-binder pocket size in propellants may play a strong role in propellant deflagration. Experimental limits on both types of burners as analog devices are established. In some burning regimes the loose-granule burner suffers from interstitial burning and the porous-plug burner deflagration is influenced by the granule-bonding technique used. This latter fact forces re-examination of the work of others in using such burners to infer stoichiometrically-correct mixture ratios from deflagration rate-mixture ratio burning characteristics.

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14. KEY WORDS	LINK A		LINK 8		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Ammonium Perchlorate Analog Techniques Combustion Mechanism Composite Propellants Particle Size Effects Polystyreme Solid Propellant Combustion						

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